IS 10532-4 (1983): Fire-Resistant Hydraulic Fluids, Part 4: Phosphate Esters Type [PCD 3: Petroleum, Lubricants and their Related Products]

BLANK PAGE

REAFFIRMED 2005 | IS : 10532 (Part 4) - 1983
(Reaffirmed 2004)

Indian Standard

SPECIFICATION FOR FIRE RESISTANT HYDRAULIC FLUIDS

PART 4 PHOSPHATE ESTERS TYPE

(First Reprint OCTOBER 2004)

UDC 621.892.099.2 : 620.193.5

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5

July 1983

AMENDMENT NO. 1 JULY 1988

TO

IS:10532(Part 4)-1983 SPECIFICATION FOR FIRE-RESISTANT HYDRAULIC FLUIDS

PART 4 PHOSPHATE ESTER TYPE

(Page 7, clause 5.1) - Add the following new clause after 5.1:

'5.2 Criteria for Conformity

5.2.1 The lot shall be declared as conforming to the requirements of the specification if all the test results on the composite sample meet the relevant specification requirements.'

(PCDC 4)

Printed at Prabhat Offset Press, New Delhi-2

AMENDMENT NO. 2 JULY 1996
TO
IS 10532 (Part 4) : 1983 SPECIFICATION FOR FIRE-RESISTANT HYDRAULIC FLUIDS
PART 4 PHOSPHATE ESTERS TYPE

[*Page* 5, *Table* 1, *Sl No.* (vi), *col* 3 *and* 5] — Substitute the following for the existing:

(3)	(5)
'$-21^{\circ}C$	$-6^{\circ}C$ '

(PCD 4)

Printed at Prabhat Offset Press, New Delhi-2

Indian Standard

SPECIFICATION FOR FIRE-RESISTANT HYDRAULIC FLUIDS

PART 4 PHOSPHATE ESTERS TYPE

Lubricants and Related Products Sectional Committee, PCDC 4

Vice-Chairman	*Representing*
SHRI P. K. GOEL	Indian Oil Corporation Ltd (Research & Development Centre), Faridabad
Members	
SHRI A. K. ARORA	Steel Authority of India Limited (Rourkela Steel Plant), Rourkela
DR N. T. BADDI	Association of Manufacturers of Petroleum Specialities, Bombay
SHRI R. CHAKRAVARTY (*Alternate*)	
SHRI M. BHADRA	Inter-Plant Standardization in Steel Industry, Subcommittee on Oils and Lubricants, IPSS 1 : 9
SHRI P. M BHAMBI	Indian Institute of Petroleum (CSIR), Dehra Dun
SHRI SUDHIR SINGHAL (*Alternate*)	
SHRI M. K. BHARGAVA	Petroleum Re-refiners Association of India, Madras
SHRI SHANTILAL DAYA (*Alternate*)	
SHRI S. R. BHATNAGAR	Indrol Lubricants & Specialities Ltd, Bombay
SHRI N. SETHURAMAN (*Alternate*)	
DR K. S. BHATTACHARAYA	Ministry of Defence (DGI)
SHRI A. K. SINHA (*Alternate*)	
SHRI B. K. CHAPRA	Central Institute of Road Transport (Training & Research), Pune
SHRI M. H. NARURKAR (*Alternate*)	
DR D. K. DAS	National Test House, Calcutta
SHRI N. C. CHATTERJEE (*Alternate*)	
DEPUTY DIRECTOR (CHEM), RDSO LUCKNOW	Railway Board (Ministry of Railways)
ASSISTANT RESEARCH OFFICER (CHEM-I) (*Alternate*)	
SHRI G. C. GOSWAMI	Assam Oil Division, Bombay
SHRI I. CHANDRA (*Alternate*)	
SHRI J. D. HARIDAS	Tata Engineering & Locomotive Co Ltd, Jamshedpur
SHRI B. GOSWAMI (*Alternate*)	

(*Continued on page* 2)

(*Continued from page* 1)

Members	*Representing*
DR G. JAYARAMA RAO	Ministry of Petroleum, Chemicals & Fertilizers
SHRI T. P. SUBRAHMANYAM (*Alternate*)	
SHRI O. K. JUNEJA	Bharat Petroleum Corporation Ltd, Bombay
SHRI I. M. MERCHANT (*Alternate*)	
SHRI S. K. KALE	Steel Authority of India Limited (Bhilai Steel Plant), Bhilai
SHRI J. L. GROVER (*Alternate*)	
SHRI V. LOBO	Oil Co-ordination Committee, New Delhi
SHRI S. N. MATHUR (*Alternate*)	
LT GEORGE MATHEW	Naval Headquarters, New Delhi
SHRI S. R. MEHTA	Hindustan Petroleum Corporation Ltd, Bombay
SHRI P. M. RAO (*Alternate*)	
SHRI R. MUKHERJEE	Indian Oil Blending Ltd, Bombay
SHRI M. J. SHAH (*Alternate*)	
SHRI S. K. NAMBIAR	Indian Oil Corporation Ltd (Marketing Division), Bombay
SHRI B. SANGHAVI (*Alternate*)	
SHRI RAJINDER SINGH	Indian Oil Corporation Ltd (Refineries Pipelines Division), New Delhi
SHRI D. K. DAS GUPTA (*Alternate*)	
SHRI T. R. RAMAKRISHNAN	The Tata Iron & Steel Co Ltd, Jamshedpur
SHRI R. A. RAO	Lubrizol India Ltd, Bombay
SHRI K. L. MALIK (*Alternate*)	
SHRI S. V. SASTRY	The Automotive Research Association of India, Pune
SHRI B. GHOSH (*Alternate*)	
SHRI P. SATYANARAYANAN	Development Commissioner, Small Scale Industries, New Delhi
SHRI N. N. SAVANT	Petrosil Oil Co Ltd, Bombay
SHRI M. A. PAES (*Alternate*)	
SHRI D. K. SINGH	Bharat Heavy Electricals Ltd, Bhopal
SHRI R. K. GUPTA (*Alternate* I)	
SHRI T. V. SUNDERRAJAN (*Alternate* II)	
CDR K. C. SINHA	Ministry of Defence (R & D)
SHRI RAJENDRA PRASAD (*Alternate*)	
DR R. D. SRIVASTAVA	Ministry of Defence (IMS & RDE)
DR P. S. VENKATARAMANI (*Alternate*)	
SHRI T. V. VARGHESE	Madras Refineries Ltd, Madras
SHRI G. SAMBAMURTHY (*Alternate*)	
SHRI M. S. SAXENA, Director (P & C)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI M. A. U. KHAN
Deputy Director (P & C), ISI

Turbine, Compressor and Hydraulic Oils Subcommittee, PCDC 4 : 11

Convener

SHRI K. S. ANAND	Indian Oil Corporation Ltd (Research & Development Centre), Faridabad

Members

SHRI B. RAMACHANDRAN (*Alternate* to Shri K. S. Anand)

(*Continued on page* 11)

Indian Standard

SPECIFICATION FOR FIRE-RESISTANT HYDRAULIC FLUIDS

PART 4 PHOSPHATE ESTERS TYPE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 12 April 1983, after the draft finalized by the Lubricants and Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 The use of fire-resistant hydraulic fluids is increasing due to a growing awareness of the dangers inherent in using mineral oils for applications where there is fire risk. There are four types of fire-resistant hydraulic fluids (FRHF), namely, dilute emulsions, invert emulsions, water glycols and synthetic fluids. In synthetic fluids, FRHF based on phosphate esters are more commonly used. These can be straight synthetic fluids in pure phosphate esters or synthetic based fluids compounded with hydrocarbon fractions. They have high film strength and good lubrication performance. Because of high thermal stability, they can operate at temperatures up to 150°C depending upon the system design. However, non-metal components of hydraulic systems, such as seals, hoses and paints commonly used in petroleum oil systems, generally speaking, are not compatible with these synthetic fluids. Special seals and others are, therefore, required to be used in systems using these fluids.

0.3 Other parts of the standard published so far are as follows:

(Part 1)-1983 Dilute emulsions for powered supports

(Part 2)-1983 Invert emulsions (water-in-oil) type

(Part 3)-1983 Water glycol type

0.4 Selection and use of the fire-resistant hydraulic fluids are covered in IS : 10531-1983* while determination of their fire-resistant characteristics is given in IS : 7895-1975†.

---

*Code of practice for selection and use of fire-resistant hydraulic fluids.

†Tests for fire-resistant characteristics of hydraulic fluids used in mining machinery.

0.5 In the preparation of this standard considerable assistance has been derived from CEGB Standard No. 20811, Issue 3, August 1975, published by the Central Electricity Generating Board, U.K.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard (Part 4) prescribes the requirements and methods of sampling and tests for fire-resistant hydraulic fluids, phosphate esters type, suitable for use in hydraulic control systems.

2. GRADES

2.1 The material shall be in three viscosity grades namely VG 22, VG 32 and VG 46 designated as HF-D 22, HF-D 32 and HF-D 46.

3. REQUIREMENTS

3.1 Description — The material shall be a clear fluid, free from foreign matter, sediment and visible impurities. It shall not contain any ingredients injurious to persons using or handling it.

3.2 Composition — The material shall be a blend of phosphate esters with additives necessary for desirable antioxidant, antirust and antifoaming properties.

3.3 The material shall also comply with the requirements prescribed in Table 1, when tested according to the appropriate methods specified in col 6 and 7 of the table.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in suitable containers as agreed to between the purchaser and the supplier. Galvanized drums/ barrels shall not be used for packing these fluids.

4.2 Marking — The containers shall be securely closed and marked with the name of manufacturer; name, type, grade and mass of the material; recognized trade-mark, if any; and with identification in code or otherwise to enable the lot of consignment or manufacture to be traced back; and the instructions for use.

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR FIRE-RESISTANT HYDRAULIC FLUIDS — PHOSPHATE ESTERS TYPE

(*Clause* 3.3)

Sl No.	Characteristics	Requirements			Method of Test, Ref to	
		Grade HF-D 22	Grade HF-D 32	Grade HF-D 46	Appendix	(P :) of IS : 1448*
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Flash point, (Cleveland open cup) °C, *Min*	180	180	180	—	P : 69
ii)	Fire point, (Cleveland open cup) °C, *Min*	290	290	290	—	P : 69
iii)	Auto-ignition temperature, °C, *Min*	580	530	470	—	P : 87
iv)	Relative density at 15/15°C, *Max*	1·25	1·25	1·25	—	P : 32
v)	Kinematic viscosity, cSt :				—	P : 25
	a) At 40°C	19·8 to 24·2	28·8 to 35·2	41·4 to 50·6		
	b) At 0°C, *Max*	300	850	1 500		
	c) At 100°C, *Min*	2·5	3·0	4·0		
	d) Minimum pumping temperature, °C, *Max* (*see* Note)	−5	0	5		
vi)	Pour point, °C, *Max*	−20	−12	−7	—	P : 10
vii)	Foaming characteristics :				—	P : 67
	a) *Foaming tendency*					
	1) Foam volume, ml, at 24°C, *Max*	150	150	300		
	2) Foam volume, ml, at 93°C, *Max*	25	25	50		
	3) Foam volume, ml, at 24°C, after test at 93°C, *Max*	150	150	300		

(*Continued*)

TABLE 1 REQUIREMENTS FOR FIRE-RESISTANT HYDRAULIC FLUIDS — PHOSPHATE ESTERS TYPE — *Contd*

Sl No.	Characteristics	Requirements			Method of Test, Ref to	
		Grade HF-D 22	Grade HF-D 32	Grade HF-D 46	Appendix	(P :) of IS : 1448*
(1)	(2)	(3)	(4)	(5)	(6)	(7)
	b) *Foam stability*				—	P : 67
	1) Foam volume, ml, at 24°C, *Max*	Nil	Nil	10		
	2) Foam volume, ml, at 93°C, *Max*	Nil	Nil	10		
	3) Foam volume, ml, at 24°C after test at 93°C, *Max*	Nil	Nil	10		
viii)	Demulsification number, seconds, *Max*	300	300	600	—	P : 95
ix)	Air release properties, minutes to 0·2 percent volume air content at 50°C, *Max*	5	10	10	—	P : 102
x)	Total acidity, mg, KOH/g, *Max*	0·2	0·2	0·2	—	P : 2
xi)	Stability tests :				A	—
	a) *Oxidation and Thermal*					
	1) Total oxidation products (TOP), percent, *Max*	0·5	0·3	0·3		
	2) Sludge, percent, of TOP, *Max*	50	50	50		
	b) *Hydrolytic and Corrosion*					
	1) Total oxidation, products, (TOP), percent, *Max*	1·0	0·8	0·8		
	2) Sludge, percent of TOP, *Max*	50	50	50		

(*Continued*)

TABLE 1 REQUIREMENTS FOR FIRE-RESISTANT HYDRAULIC FLUIDS — PHOSPHATE ESTERS TYPE — *Contd*

Sl No.	Characteristics	Requirements			Method of Test, Ref to	
		Grade HF-D 22	Grade HF-D 32	Grade HF-D 46	Appendix	(P :) of IS : 1448*
(1)	(2)	(3)	(4)	(5)	(6)	(7)
	3) Corrosion, mg, *Max*	5·0	5·0	5·0		
xii)	Rust preventing characteristics	Shall pass the test after 20 h			—	P : 96 (Method A)
xiii)	Water content, percent by volume, *Max*	0·1	0·1	0·1	—	P : 40
xiv)	Four ball test, 40 kg, 1 800 rev/min, 55 ± 2° for 1 h	← Scar dia 0·5 mm, *Max* →			—	P : †
xv)	Fire-resistant characteristics	Shall pass the test A, B and C			IS : 7895-1975‡	

NOTE — Minimum pumping temperature corresponds to viscosity of the oil in the pump, equivalent to 850 cSt.

*Methods of test for petroleum and its products.
†Under preparation. Till such time ASTM D 2266-67 may be followed.
‡Test for fire-resistant characteristics of hydraulic fluids used in mining machinery.

4.2.1 The product may also be marked with Standard mark.

4.2.2 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS : 1447-1966*.

*Methods of sampling of petroleum and its products.

6. STORAGE

6.1 The material shall be stored and handled, strictly in accordance with the supplier's instructions.

7. SAFETY PRECAUTIONS

7.1 While handling these fluids the following safety precautions shall be observed:

a) The fluid does not cause skin irritation ordinarily but wearing of protective clothing is recommended.

b) Continued exposure to the fumes should be avoided. Breathing vapours from heated or burning product should be avoided.

c) Smoking, eating or drinking shall be prohibited when these fluids are being handled.

APPENDIX A

[*Table* 1, *Item* (xi)]

TEST FOR OXIDATION AND HYDROLYTIC STABILITY AND CORROSION POTENTIAL

A-1. SCOPE

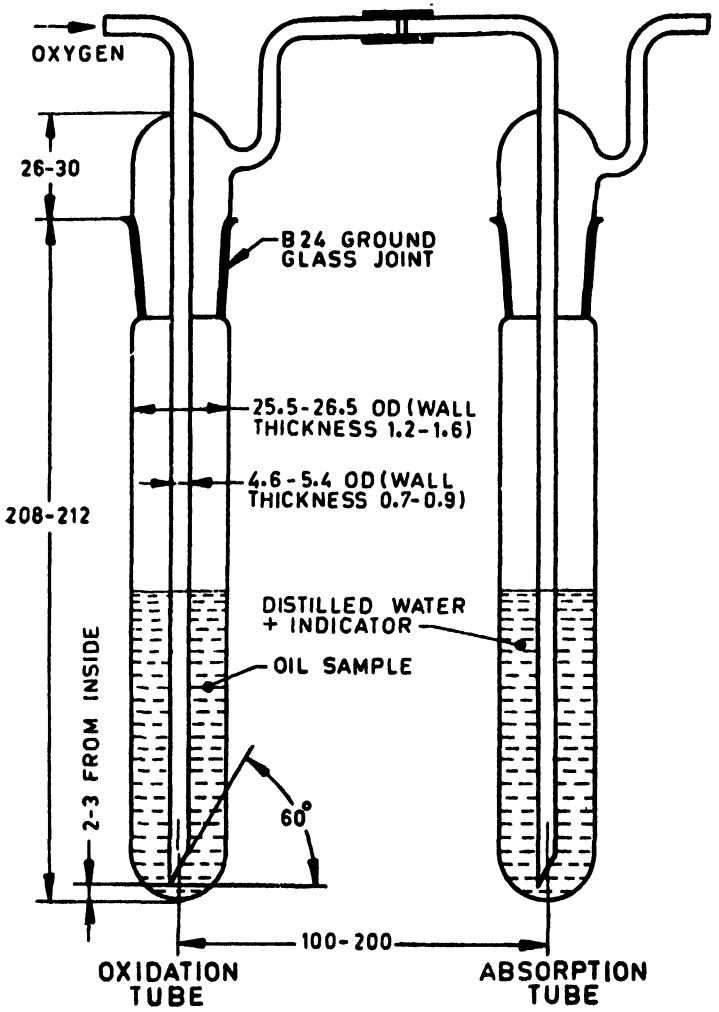
A-1.1 This method is comparatively short test to measure the resistance to oxidation and hydrolysis under specified conditions of new phosphate ester fire-resistant fluids. It also measures the corrosivity of the fluid and its degradation products in the presence of water.

A-2. OUTLINE OF THE METHOD

A-2.1 Oxygen is passed for 164 h through a sample *A* of the fluid, and another sample *B* of the fluid with added solid metal catalysts (iron and copper) and water whilst maintained at 120°C. The volatile acid products, the acidity of the fluid; the sludge and the sludge insoluble in organic solvents are determined, where sample *A* is used for oxidation and thermal stability, and sample *B* for hydrolytic stability and corrosivity.

A-3. APPARATUS

A-3.1 Oxidation Tubes — Manufactured from borosilicate or neutral glass having the dimensions shown in Fig. 1.



All dimensions in millimetres.

FIG. 1 OXIDATION AND ABSORPTION TUBES

A-3.2 Heating Bath — An aluminium alloy block heater or oil bath thermostatically controlled to maintain the oil in the desired number of oxidation tubes at the required temperature of 120 ± 0·5°C (*see* Fig. 2). This temperature shall be read on a thermometer type IP 81C inserted in a test tube to within 5 mm from the bottom; this test tube shall be filled with oil up to the immersion line of the thermometer and placed in the heating bath. The temperature of the upper surface shall be kept at 60 ± 5°C. Measure this temperature by the use of a thermometer in a drilled aluminium block (*see* Fig. 2); the surfaces of this block, other than that against the upper surface of the heating bath, are protected by suitable insulation. This block should be placed as near to the holes as practicable and within the area of the aluminium heater block. When using an aluminium block heater the test tubes are inserted into the holes to an overall depth of 150 mm. The depth of the holes in the heating part of the block shall be at least 125 mm and short metal collars, passing through the insulating cover and surrounding each oxidation tube, will ensure heating over the 150 mm length of the tube. In the case of oil baths the oxidation tubes shall be immersed to a depth of 137 mm in the oil and to an overall depth of 150 mm in the bath. For both types of heating bath the height of the oxidation tubes above the upper surface shall be 60 mm and the diameter of the holes shall be just sufficient to allow insertion of the specified tube. In case of a slackness a 25 mm diameter O-ring may be placed round the tube and pressed against the heater surface.

A-3.3 Filtering Crucibles — Gooch type crucibles with sintered filter disc of Grade 4 porosity (5 to 15 microns) 35 ml capacity.

A-3.4 Porcelain Crucibles — 50 ml capacity.

A-3.5 Soap Bubble Flowmeter — for checking the oxygen flow rate (*see* Fig. 3).

A-3.6 Burette — volume 10 ml with graduations of 0·01 ml.

A-3.7 Thermometers — conforming to specifications IP 81C and IP 15C, or others of suitable range and equal or greater accuracy.

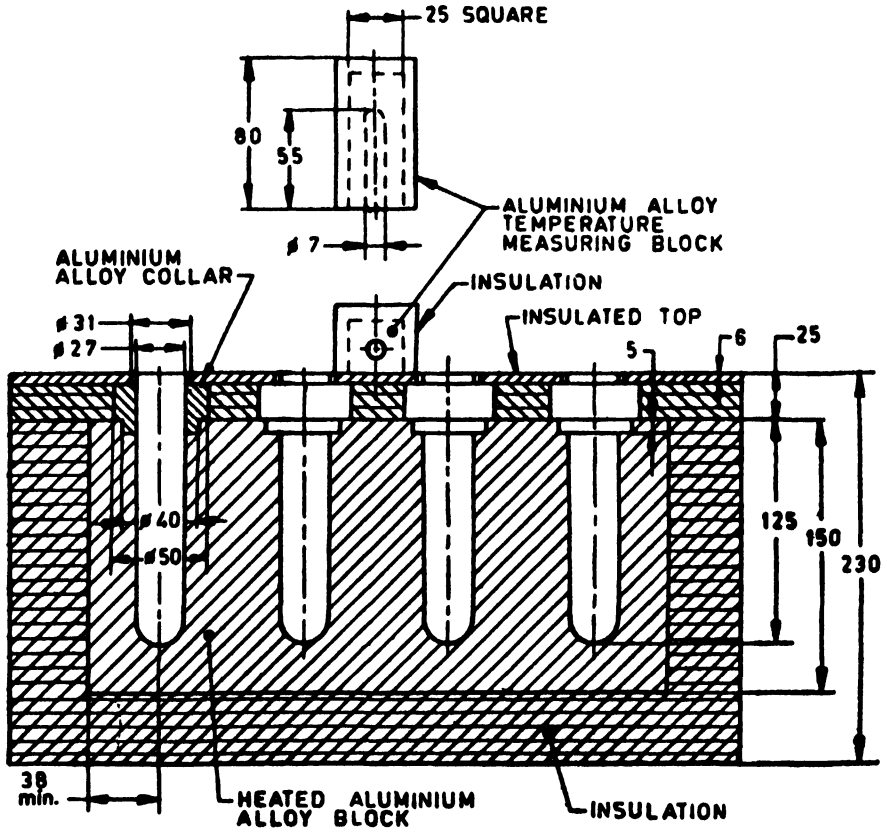
A-3.8 Absorption Tubes — These are similar to the oxidation tubes (*see* **A-3.1**) but with the bubbling tube modified as in Fig. 4.

A-4. REAGENTS AND MATERIALS

A-4.1 Catalysts

a) *Copper Wire* — 1·04 ± 0·01 mm diameter, annealed, plain.

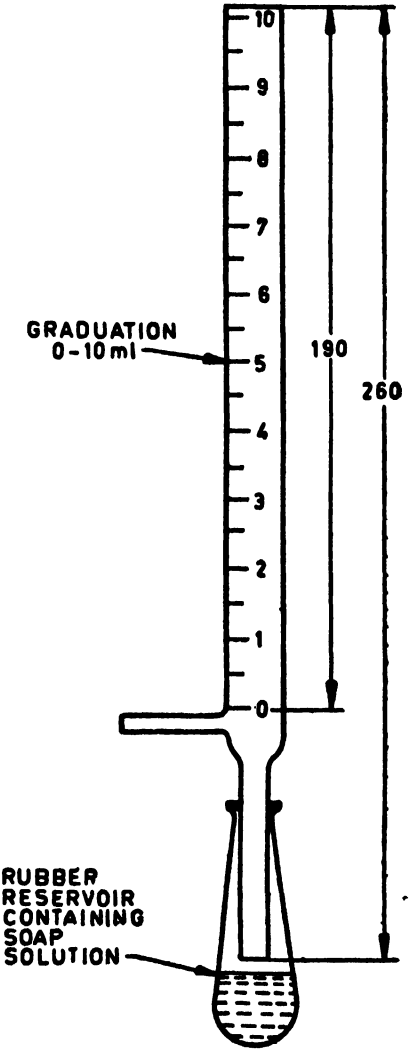
b) *Low Metalloid Steel Wire* (*Thermocouple Quality*) — 1 mm diameter.



All dimensions in millimetres.

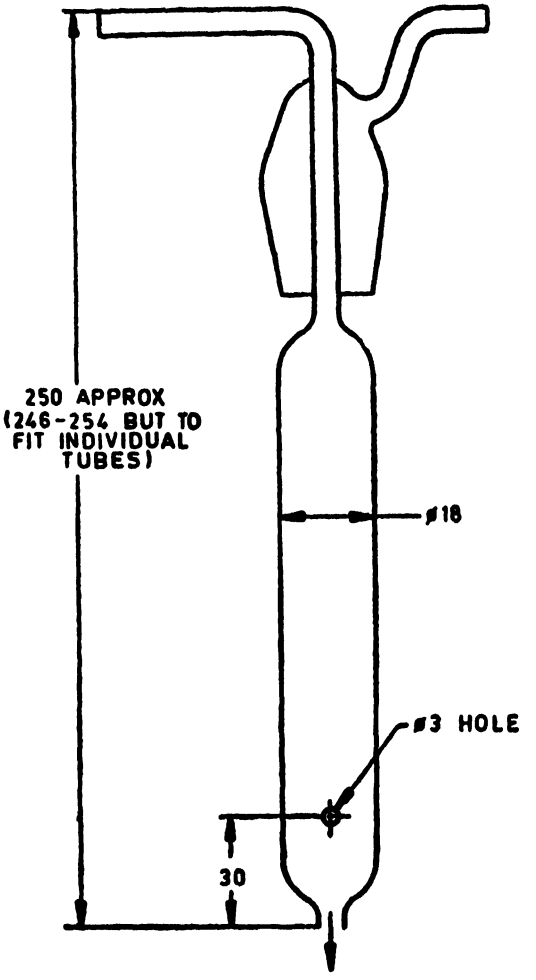
FIG. 2 TYPICAL METAL HEATING BATH

A-4.2 Oxygen — commercial product obtained from liquid air (minimum purity 99·4 percent). The oxygen shall be dried by passing through a suitable desiccant. A 10-litre flask, acting as a surge vessel, smooths the oxygen flow, excess of which bubbles through mineral oil contained in a test tube. The method used to regulate the oxygen flow is left to the discretion of the operator, but the flow rate shall be checked by use of the soap bubble flowmeter (*see* Fig. 3) connected after the absorption vessel.



All dimensions in millimetres.

FIG. 3 SOAP BUBBLE FLOWMETER



All dimensions in millimetres.

All other dimensions to Fig. 1.

FIG. 4 ABSORPTION TUBE BUBBLER FOR MODIFIED TEST

A-4.3 Alkali Blue Solution — 2 g/100 ml.

A-4.4 Phenolphthalein Solution — 1 g/100 ml alcoholic solution.

A-4.5 *n*-Heptane — same as that used as a reference fuel in determining the octane number of gasoline.

A-4.6 Hydrochloric Acid — 0·1 N aqueous solution.

A-4.7 Potassium Hydroxide — 0·1 N alcoholic solution.

A-4.8 Toluene — pure, sulphur-free.

A-4.9 Chloroform

A-4.10 *Iso*propyl Alcohol

A-4.11 Methyl Alcohol

A-5. PREPARATION OF APPARATUS

A-5.1 Cleaning the Test Tubes — The oxidation and absorption tubes shall be chemically cleaned. A satisfactory method of cleaning is to wash with acetone, followed by distilled water. Drain and then soak in concentrated sulphuric acid for a minimum of 16 hours. Drain and complete removal of the acid by washing, first with tap water, then with distilled water. Dry the tubes in an air oven at 105 to 110°C for at least 3 hours, and then allow them to cool to room temperature in a desiccator in which they are kept until they are used.

A-6. PROCEDURE

A-6.1 Weigh sample *A* 25 g and sample *B* 10 ± 0·5 g of the fluid into two oxidation tubes respectively. Clean 300 mm each of the copper and iron wire with absorbent cloth wet with *n*-heptane, followed by abrasion with No. 100(00) silicon carbide cloth until a fresh metal surface is exposed. Wipe with dry absorbent cotton until all loose particles of abrasive and metal have been removed. In subsequent operations handle the wires with absorbent cotton (gloves) or tweezers to prevent them coming into contact with the skin. Wind the wires simultaneously alongside each other on a threaded mandrel. Lift the ends of the wires from the slot in the end of the mandrel and twist together for approximately three turns. Adjust the length of the coil to 20 mm. Store in petroleum spirit until insertion in tube *B*. Put tubes *A* and *B* into the heating bath at 120 ± 0·5°C.

A-6.2 Connect the oxidation tubes to the absorption tubes into which has been placed 25 ml of neutral distilled or deionized water and 5 to 6 drops of phenolphthalein solution (*see* Note).

NOTE — To avoid evaporation of water the absorption tube shall be protected from the heating bath by insulation.

A-6.3 Connect and adjust the oxygen flow at 1·0 ± 0·1 l/h which shall be checked daily. After 1 hour slightly raise the drechsel head of tube *B* and add 0·5 ml distilled water, from a fine bore pipette, through the gap between the socket and cone. Repeat the addition of water after 24, 48, 72 and 96 hours.

A-6.4 After 164 hours, stop the oxygen flow, disconnect the oxidation and absorption tubes and remove the oxidation tubes from the bath.

A-6.5 Treat the absorption tubes as follows.

A-6.5.1 *Volatile Acids*

A-6.5.1.1 As quickly as possible after the test, titrate the water in the absorption tube with the alcoholic potassium hydroxide solution.

A-6.6 Treat the oxidation tube as follows.

A-6.6.1 *Sludge Determination* — Cool the sample of 25 g of artificially aged oil in the dark for 1 hour and then pour it into a conical flask of 500 ml capacity, fitted with a ground glass stopper. Use 300 ml a mixture of equal volumes of *n*-heptane (*see* Note) and toluene to recover the oil adhering to the test tube and oxygen lead-in tube and add the washings to the oil in the flask.

NOTE — If the normal heptane recovered from previous test is used it shall be acid-free and comply with the original specification (*see* A-4.5).

A-6.6.1.1 Allow the mixture to stand in the dark for 24 hours, at a temperature of 20 ± 2°C, then filter through the filtering crucible previously dried to constant mass.

A-6.6.1.2 At the start of filtering only a small pressure drop should be used to prevent the sludge passing through the filter. Cloudy filtrates should be passed through a second time.

A-6.6.1.3 Carefully remove all traces of oil by repeated washing of the sludge with a mixture of equal volumes of normal heptane and toluene. The total volume of the solvent used for the washing of the sludge shall be 150 ml. Dry the crucible containing the sludge at 110°C to constant mass. The filtrate shall be used for the determination of soluble acidity.

A-6.6.1.4 Dissolve any sludge adhering to the test tube and to the oxygen lead in tube in small quantities of chloroform (a total of 30 ml) and transfer the solution into a tared porcelain crucible. After the evaporation of the chloroform dry at 110°C to constant mass.

A-6.6.2 *Soluble Acidity* — Collect the heptane/toluene solution obtained after filtering off the sludge in a 500-ml measuring flask and make up to the mark with a mixture of equal volumes of *n*-heptane and toluene. Make three determinations of the neutralization value on 100 ml samples of the *n*-heptane/toluene/oil solution.

A-6.6.2.1 Immediately before use prepare the titration solvent as follows:

Add 2 ml of the alkali blue solution to 100 ml of a mixture of 60 ml of toluene and 40 ml of isopropyl alcohol containing 5 percent of water. Neutralize the mixture of 60 ml alcoholic potassium hydroxide solution to give a red colour comparable to that of a 10 percent solution of cobalt nitrate $Co(NO_3)_2\ 6H_2O$, and this colour shall persist for at least 15 seconds. Add this neutralized solvent with swirling, to 100 ml of the heptane/toluene solution then titrate with the alcoholic potassium hydroxide at a temperature not exceeding 30°C.

A-6.7 Insoluble and Inorganic Material from Tube (*B*)

A-6.7.1 After drying and weighing the sludge in the filter and porcelain crucibles from tube (*B*) proceed as follows:

Extract the sludge in the porcelain crucible with successive quantities of warm chloroform and methyl alcohol, approximately 50 ml of each. Transfer to the filter crucible and vacuum filter. Dry the filter crucible for 1 hour at 105 to 110°C. Cool for 1 hour and weigh. Note that to repeat results for low sludge and insolubles contents (for example $< 0{\cdot}02$ percent), clean solvents shall be used. Whilst solvents are normally sufficiently clean it may be advisable to pre-filter all solvents through a No. 4 filter disc.

A-7. CALCULATION AND REPORT

A-7.1 Volatile acidity (VA, mg KOH/g) $= \dfrac{A \times 56{\cdot}1 \times N}{25}$ for tube (*A*)

and $= \dfrac{A \times 56{\cdot}1 \times N}{10}$ for tube (*B*)

A-7.2 Soluble acidity (SA, mg KOH/g) $= \dfrac{A \times 56{\cdot}1 \times N}{5}$ for tube (*A*)

and $= \dfrac{A \times 56{\cdot}1 \times N}{2}$ for tube (*B*)

where

A = the volume of 0·1 N alcoholic potassium hydroxide solution necessary to neutralize the *n*-heptane/toluene solution, and

N = the normality of the alcoholic KOH solution used.

A-7.3 Total acidity (TA) = VA + SA

A-7.4 Total sludge (D percent) = ($a + b$) × 4 for tube (A)

and = ($a + b$) × 10 for tube (B)

where

a = the mass of sludge insoluble in toluene/*n*-heptane in grams, and

b = the mass of sludge recovered by chloroform in grams.

A-7.5 Total oxidation products (TOP percent) $= D + \frac{\text{TA} \times 100 \times 0{\cdot}18}{1\,000 \times 0{\cdot}056\,1}$

A-7.6 Insoluble and inorganic material for tube B = mass of material insolube in chloroform and methyl alcohol in grams.

A-8. PRECISION

A-8.0 An indication of the precision of this method can be obtained from the following (within laboratory reproducibility).

A-8.1 Oxidation and Thermal Stability

A-8.1.1 *Total Oxidation Products* — Duplicate results obtained by the same operator in the same laboratory on tests carried out at different times should be considered suspect if they differ by more than 28 percent of their mean for results in the range 0·05 to 3·0 percent.

A-8.2 Hydrolytic Stability

A-8.2.1 *Total Oxidation Products* — Duplicate results obtained by the same operator in the same laboratory on tests carried out at different times should be considered suspect if they differ by more than 28 percent of their mean for results in the range 0·13 to 3 percent.

A-8.2.2 *Corrosion* — Duplicate results obtained by the same operator in the same laboratory on tests carried out at different time should be considered suspect if they differ by more than 112 percent of their mean for results in the range 0·0 to 5·0 mg insoluble and inorganic deposits.

(*Continued from page* 2)

Members	*Representing*
SHRI A. K. ARORA	Steel Authority of India Limited (Rourkela Steel Plant), Rourkela
SHRI M. K. BHARGAVA	Petroleum Re-refiners Association of India, Madras
SHRI SHANTILAL DAYA (*Alternate*)	
SHRI S. R. BHATNAGAR	Indrol Lubricants & Specialities Ltd, Bombay
SHRI N. SETHURAMAN (*Alternate*)	
SHRI B. K. CHAPRA	Central Institute of Road Transport (Training & Research), Pune
SHRI M. H. NABURKAR (*Alternate*)	
SHRI N. C. CHATTERJEE	National Test House, Calcutta
SHRI S. A. DHIR	Lubrizol India Ltd, Bombay
SHRI R. A. RAO (*Alternate*)	
SHRI O. K. JUNEJA	Bharat Petroleum Corporation Ltd, Bombay
SHRI G. BALAKRISHNAN (*Alternate*)	
SHRI A. N. KARMARKAR	Automobile Products of India Ltd, Bombay
SHRI S. R. MEHTA	Hindustan Petroleum Corporation Ltd, Bombay
SHRI P. KRISHNAMOORTHY (*Alternate*)	
SHRI D. M. MERCHANT	Bhavnagar Oil & Chemical Industries Pvt Ltd, Bombay
SHRI A. F. J. BAPLIST (*Alternate*)	
SHRI R. MUKHERJI	Indian Oil Blending Ltd, Bombay
SHRI M. J. SHAH (*Alternate*)	
SHRI P. N. PATEL	Association of Manufacturers of Petroleum Specialities, Bombay
SHRI K. K. NEYOGI (*Alternate*)	
SHRI V. RAMACHANDRAN	Brakes India Ltd, Madras
SHRI P. KRISHNAMOORTHY (*Alternate*)	
REPRESENTATIVE	Directorate General of Mines Safety, Dhanbad
SHRI B. SANGHAVI	Indian Oil Corporation Ltd (Marketing Division), Bombay
SHRI B. LAXMINARAYANAN (*Alternate*)	
SHRI N. N. SAVANT	Petrosil Oil Co Ltd, Bombay
SHRI M. A. PAES (*Alternate*)	
DR A. SETHURAMIAH	Indian Institute of Petroleum (CSIR), Dehra Dun
SHRI S. K. JAIN (*Alternate*)	
SHRI K. B. SHRIVASTAVA	Tide Water Oil Co (India) Ltd, Calcutta
SHRI J. NAIK (*Alternate* I)	
SHRI N. C. MITTAL (*Alternate* II)	
SHRI D. K. SINGH	Bharat Heavy Electricals Ltd, Bhopal
SHRI G. ACHUTARAMAIAH (*Alternate* I)	
SHRI B. S. SRIVASTAVA (*Alternate* II)	
SHRI G. P. SINGH	Steel Authority of India Limited (Bokaro Steel Plant), Bokaro Steel City
SHRI A. K. MISRA (*Alternate*)	
SHRI J. P. SIRPAL	Naval Headquarters, New Delhi
SHRI A. P. GOEL (*Alternate*)	
DR R. D. SRIVASTAVA	Ministry of Defence (DGI)
SHRI RAJENDRA SINGH (*Alternate*)	
SHRI T .V. VARGHESE	Madras Refineries Ltd, Madras
SHRI P. C. D. G. SAMUEL (*Alternate*)	

BUREAU OF INDIAN STANDARDS

Headquarters:
Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 23230131, 23233375, 23239402 **Fax**: 91+011 23234062, 23239399, 23239382
E-mail: bis@vsnl.com website: http://www.bis.org.in

Central Laboratory:	***Telephone***
Plot No. 20/9, Site IV, Sahibabad Industrial Area, SAHIBABD 201010	2770032

Regional Offices:	
Central: Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002	23237617
*Eastern; 1/14 CIT Scheme VII M, V.I.P. Road Kankurgachi, KOLKATA 700054	23378662
Northern: SCO 335-336, Sector 34-A, Chandigarh 160022	603843
Southern: C.I.T. Campus, IV Cross Road, CHENNAI 600113	254 19 84
Western: Manakalaya, E9, MIDC, Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	2832 92 95

Branch offices:	
'Pushpak' Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	560 13 48
Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE	839 49 55
Commercial-cum-office Complex, opp. Dushera Maidan, Arera Colony, Bittan Market, BHOPAL 462016	242 34 52
62/63, Ganga Nagar, Unit VI, BHUBANESHWAR 751001	240 3139
5TH Floor, Kovai Towers, 44 Bala Sundaram Road, COIMBATORE 641018	221 0141
SCO 21, Sector 12, Faridabad 121007	2292175
Savitri Complex , 116 G.T. Road Ghaziabad 201001	2861498
53/5 Ward No. 29, R.G. Baraua Road 5 by-lane, Apurba Sinha Path GUWAHATI 781003	2541137
5-8-56C L.N. Gupta Marg, Nampally Station Road, HYBERABAD 500001	23201084
E-52, Chitranjan Marg, C-Scheme, JAIPUR 302001	2373879
117/418 B Sarvodaya Nagar, KANPUR 208005	2218774
Sethi Bhavan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	2215698
NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	2525171
Mahavir Bhavan, First Floor, Ropar Road, NALAGARH 174101	221451
Patliputra Industrial Estate, PATNA 800013	2262808
First Floor, Plot Nos 657-660, Market Yard, Gultkdi, PUNE 411037	4268659
"Sahajanand House" 3rd Floor, Bhaktinagar Circle, 80 Feet Road, RAJKOT 360002	2378251
T.C. No. 14/1421, University P.O. Palayam, THIRUVANANTHAPURAM 695034	2322104
1st Floor, Udyog Bhavan, VUDA, Siripuram Junction, VISHAKHAPATNAM-03	2712833

Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, Kolkata 700072	22371085
Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	23096528

Printed at Prabhat Offset Press, New Delhi-2